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Characterization by thermal analysis of natural kieselguhr and sand for industrial application

Hazem Meradi[a*], L'Hadi Atoui[b], Lynda Bahloul[a], Khaled Boubendira[a], Abdelhak Bouazdia[a], Fadhel Ismail[c]

[a]Welding and NDT Research Center (CSC). BP 64 Cheraga - Algeria
[b]Department of *Metallurgy* and Materials Engineering, University of Annaba, Algeria
[c]Department of Process Engineering, University Badji Mokhtar of Annaba, Algeria

Abstract

Kieselguhr also known diatomite is a silica-based mineral, usually light in color (white if pure). It is very finely porous and very low in density. It is composed mainly of silica as career sand. The aim of this study was to characterize the kieselguhr and sand for together use in industry for various applications. Both products come of Sig deposit (West Algeria).The results of simultaneous analyses by Thermogravimetric Analysis and Differential Scanning Calorimetry (TGA-DSC) for kieselguhr and sand sample shows almost identical peaks except for the peak which appears at 574° C which corresponds to the transformation of quartz sand that does not appear in the Kieselguhr sample because of its amorphous character and higher exothermic peak at 574° C approximately.

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*Keywords:*Kieselguhr, diatomite, sand, thermal analysis,DSC, TGA

* Corresponding author. Tel.: +(213) 661697553; fax: +(213)38875982 .
E-mail address: meradi213@yahoo.com

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1. Introduction

The kieselguhrs or diatomaceous earths ($SiO_2 \cdot nH_2O$) are composed essentially of more or less well crystallized silica, as well as a small quantity of alumina, iron oxide, and traces of other oxides. It is a natural material formed from the remains of diatoms and has many important industrial applications due to its unique properties, i.e. micronized and submicronized porous structure, high permeability, small particle size, low thermal conductivity and density and high specific surface. These applications include filter aid [1,2], functional filler, catalysis support, and carrier application [3-18], cement production [19], insulating materials [20,21] and recently, the use of kieselguhr as a possible additive for improvement of hydrogen storage properties of MgH_2 has also been reported [22]. The kieselguhr and sand reserve in Algeria is estimated at several million tons and it is located in Sig region (from 50 km of Mascara, west of Algeria).The aim of this study was to characterize the kieselguhr and sand to their use together in various industrial application such as filtration, insulation, sensor. Characteristics of kieselguhr in different countries are shown in the Table 1 [23-24]. The main limitations of kieselguhr are, reduction in the flowability of grain, reduction of the bulk density of grain, ineffective in some situations, discomfort due to air borne dust and health concerns due to crystalline silica [25]. Diatomaceous materials and sand from Deposit Sig, Algeria were microscopically investigated to determine their characteristics, ways of processing and their specifications for industrial applications.

Table 1. Characteristics of kieselguhr for different countries

properties	Kieselghur US dried	Kieselghur Danish calcined	Kieselghur US calcined	Kieselghur French calcined	Kieselghur German calcined	Kieselghur Algeria dried
Color	white gray	yellow brown	Rose	yellow brown	brown	white
pH	7	5.2	7.5	6.9	7	9.7
Surface area(m^2/g)	19.2	25.4	15.2	13	16.1	70.35
Average diameter (µm)	14.2	19.3	15.9	14.1	13.9	15.1
Permeability (cm/s)	0.06	0.09	0.28	0.09	0.08	0.02
Crystalline Quantity (%)	2	2.2	7.6	9.2	9.8	8.5
Density (g/l)	288	280	271	255	209	243

2. Experimental

In the present work the characterization of kieselguhr and sand from Sig Deposit (West Algeria) have been studied by thermal analysis to see the thermal behavior and mass loss at different temperature. The chemical composition realized by X Ray Fluorescence (XRF) is presented in Table 2 for kieselguhr sample and Table 3 for sand sample. The Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) were carried out using a fully computerized Netzsch STA 409 PC simultaneous TGA-DSC instrument. Thermal analysis focused on two samples of sand and kieselguhr. The tests were performed under the same experimental conditions, about 10 mg of product was placed into Al_2O_3 crucible for simultaneous TGA-DSC analysis and was heated at a rate of 10 °C/min, from room temperature to 1100 °C in a static air environment. The morphology of the mixed sample (50 % kieselguhr and 50 % sand) was observed using a scanning electron microscope (SEM 5600).

Table 2. Chemical composition of the kieselguhr sample.

Component	MgO	Fe_2O_3	SiO_2	TiO_2	CaO	K_2O	Al_2O_3	Na_2O
Wt(%)	2.15	1.19	73.4	0.027	13.58	0.78	3.15	0.002

Table 3. Chemical composition of the sand sample.

Component	MgO	Fe_2O_3	SiO_2	TiO_2	CaO	K_2O	Al_2O_3	Na_2O
Wt(%)	0.28	0.74	93.57	0.027	1. 87	0.59	2.12	0.48

3. Results and discussion

The chemical composition of the diatomite is as follows 79.72% SiO_2, 10.20% Al_2O_3. There are also water molecules and impurities consisting of $CaCO_3$, Fe_2O_3, TiO_2. The results of simultaneous analyses TGA-DSC for kieselguhr was presented in a previous work presented in International Conference TMREES 2015 which four mass losses and different transformation peaks are observed. For the sand sample, we can see from figure 1, that first loss (about 3.2 %) between room temperature and 600 °C, the second mass loss (about 2.63 %) from 600 °C to 800 °C, the last loss (about 0.41 %) in the range from 800 °C to1100 °C, whether a total mass loss of 6.24 % against 21.60 % for kieselguhr (figure 2).The results from simultaneous analysis DSC-TGA for sand sample indicate that when the temperature is increased, several peaks take place. These are clearly detected in the graph in figure 1. We can see from DSC spectrum three endothermic peaks at 68.9, 574.7 and 723 °C, and one exothermic peak at 935.4 °C. The endothermic peak centered at 68.9 °C was assigned to the loss of water absorbed on the sand. The peak at 574.7 °C might be due to the quartz transformation. The large endothermic peak at 723 °C has been assigned to a formation of siloxane bridges resulting from dehydroxylation of isolated silanol groups on the internal surface of the sand that corresponds to the maximum loss of mass (5.83 % against 20,96 % for kieslguhr) as show in figure 2. At 935.4 °C, we can see an exothermic peak caused by the cristalization. The exothermic direction is shown along the vertical axis. Generally, quartz is known to give an endothermic reaction between 565 and 575 °C, in figure 1, the endothermic peak at 574.7 °C is very clear because the cristalline structure of sand.It is clear that almost mass loss is maximum for the two samples at 800 °C but more important for kieselguhr (Fig.2).The thermal analysis by Differential Scanning Calorimetry for the two sample of sand and kieselguhr show almost identical shape as showed in figure 3. But we can see in this case the large endothermic peak at about 800 °C and exothermic peak at 935 °C for kieselguhr.Scanning Electron Microscope (SEM) shows porous structures with presence of impurities (Fig.4). We can note the circular form of pores and we can also see the natural kieselguhr is typical, showing dominant a granulate species. It was found to be essentially amorphous but also it contained ankerit, calcite and quartz for kieselguhr and cristobalite, calcite, microcline and quartz for sand with crystalline structure [26]. It is found that the behaviour is almost identical to different temperature but of course with a fairly large gap in the decomposition of calcite. In addition, it is clear peak at 574 °C during the sand sample heating something that does not appear in the sample diatomite because of its amorphous character.

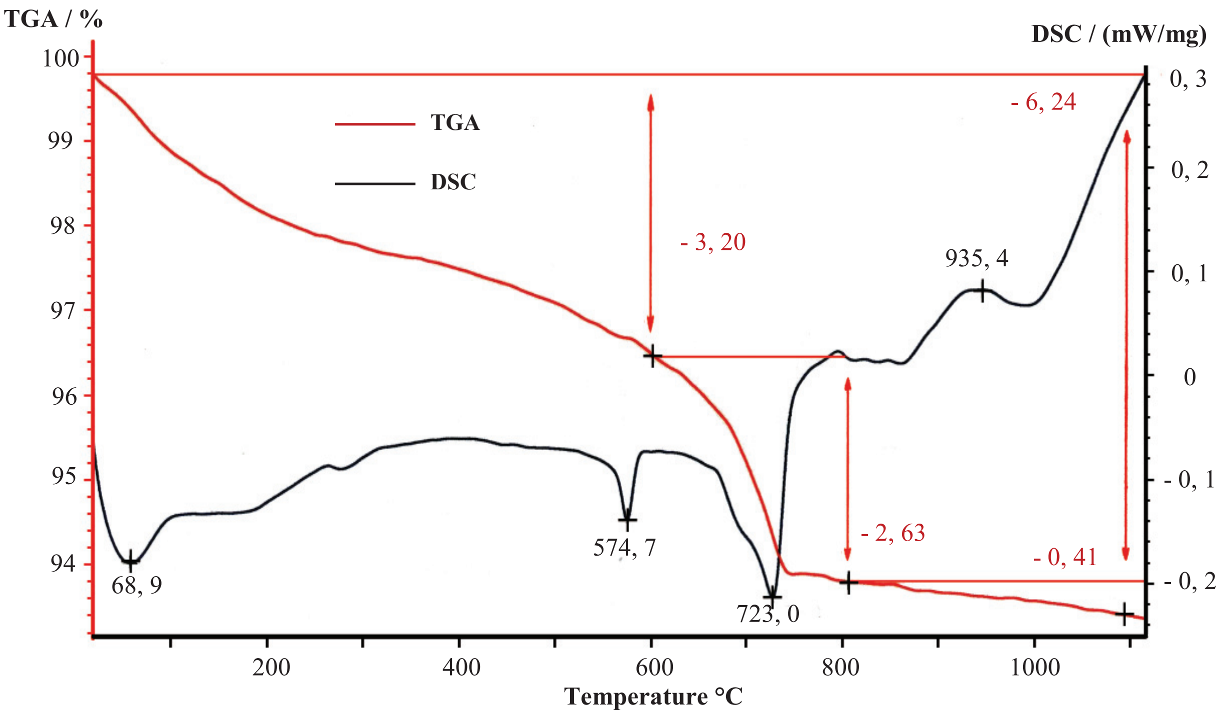

Fig 1: Results of Differential Scanning Calorimetry and Thermovimetric Analysis of sand.

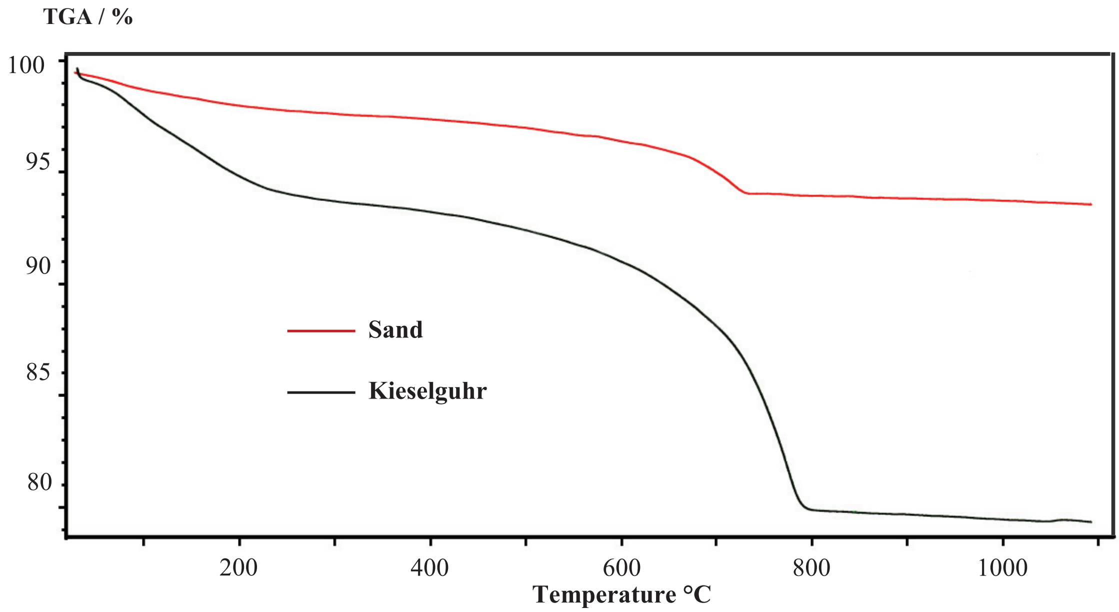

Fig 2: Mass losses of kieselguhr and sand at different temperature

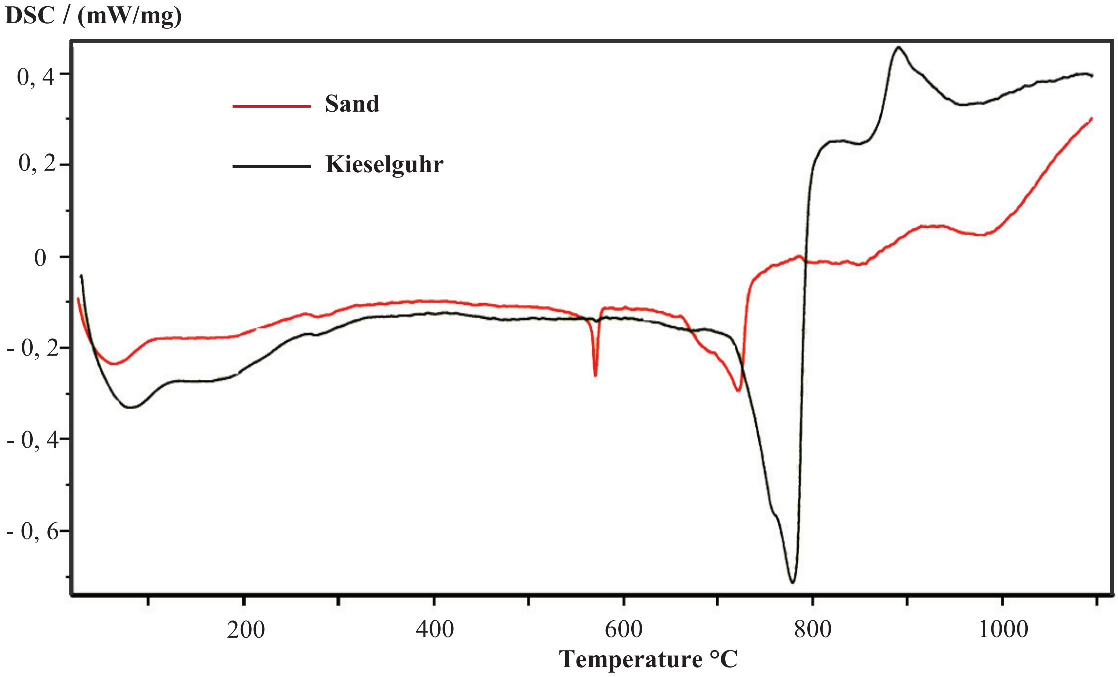

Fig 3: Result of Differential Scanning Calorimetry Analysis of kieselguhr and sand.

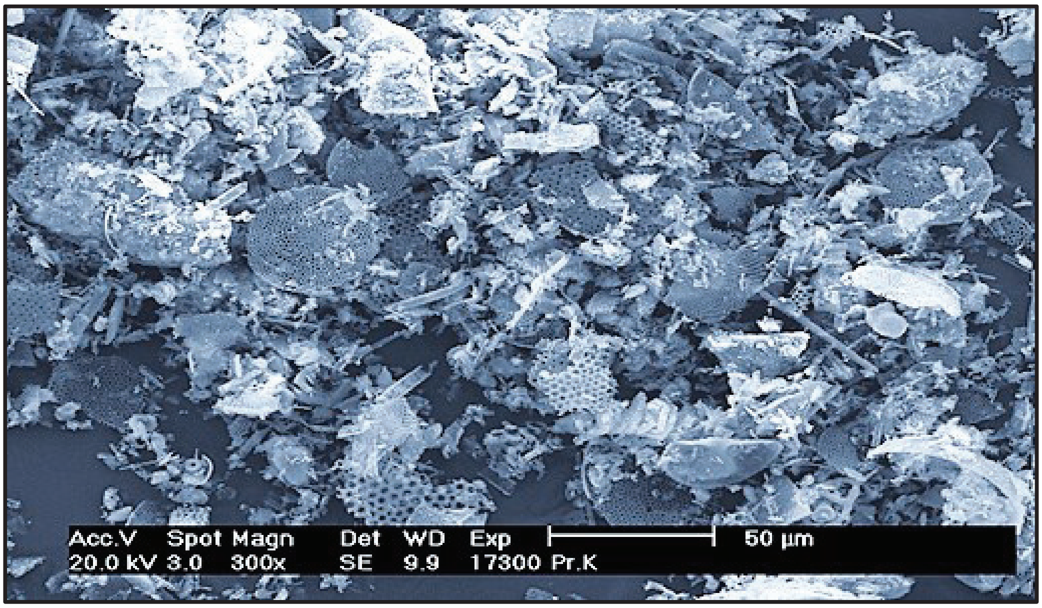

Fig.4: SEM micrography of mixed powder (kieselguhr and sand) with 3000X magnification

4. Conclusion

The results obtained showed that chemical composition of kieselguhr and sand are predominantly consists of SiO2 (amorphous and crystalline structure) with impurities additions of Fe, Al, Ca, Mg, Na, K. The CaO and MgO contents in kieselguhr are high compared to the sand sample due to the presence of carbonate minerals, The Na_2O and K_2O content are mainly attributed to the presence of feldspars and illite. Kieselguhr have a porous structure with ordered size distribution of the pores, this give low density. SEM figure of mixed sand and diatomite showed the presence of intact diatomite skeletons but with impurities and showed the presence of different skeleton forms and their morphological characteristics. Thermal analysis of kieselguhr and sand of Algeria were investigated for both to determine their characteristics, ways of processing and their specifications for various industrial applications. Thermogravimetric analysis of kieselguhr sample was depicted and the loss mass was maximal at 800 °C.The peak at 576°C analyzed by DSC showed the transformation of quartz which is more important for sand. The aim of this research is to study the way to use kieselguhr and sand with respect for environmental protection

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